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AT ELEVATED TEMPERATURES

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and H. B. Dexter

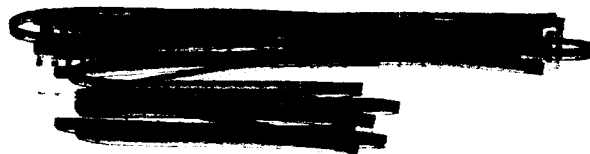
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### ABSTRACT

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In the investigation of the salt stress corrosion of Ti-8Al-1Mo-1V alloy sheet, many small self-stressed and residual stress specimens were salt coated, exposed up to 6400 hours at 400° F to 600° F (477° K to 589° K), and tested at room temperature to determine the extent of the stress corrosion cracking. Coatings of NaCl were more corrosive than CaCl<sub>2</sub>, MgCl<sub>2</sub>, or sea salt, and thin coatings were more damaging than thick. Environmental tests showed that a decrease in oxygen and air pressure reduced stress corrosion, but that the role of moisture and air velocity still remained in doubt. The temperature threshold for salt stress corrosion after 5000 hours was slightly above 400° F for a stress of 50 ksi (345 MN/m<sup>2</sup>). Short thermal cycles of 2 or 4 hours produced less corrosion than that for steady temperature conditions. Stress corrosion cracks were intergranular, and a correlation was obtained between the results of the room-temperature corrosion tests and the crack penetration. TiCl<sub>2</sub> appeared to be one of the corrosion products. Surface treatments such as shot peening, nickel plating, vibratory cleaning, and polyimide coatings proved effective in preventing corrosion for exposures up to 2000 hours at 600° F.

### INTRODUCTION

Of the various candidate structural materials for a Mach 3 supersonic transport, Ti-8Al-1Mo-1V alloy sheet in the duplex anneal condition has been favored because of structural efficiency considerations involving strength, stiffness, fracture, and fatigue (see, for example, refs. 1 and 2). Other considerations, however, such as possible metallurgical instability after prolonged elevated-temperature exposure and the susceptibility of this alloy to salt stress corrosion at elevated temperature, still require investigation (ref. 3). Because of these drawbacks, particularly the latter, extensive research has been undertaken by the aircraft industry, material producers, and research laboratories to determine the severity and extent of the corrosion problem and the nature of the phenomenon.

An investigation of the salt stress corrosion of titanium alloy and stainless-steel sheet materials at elevated temperatures was begun about 3 years ago at the NASA Langley Research Center. Results obtained so far include the relative susceptibility of four titanium alloys (ref. 4), the effect of environmental factors on Ti-8Al-1Mo-1V (ref. 5), the susceptibility of six stainless

steels at both ambient and elevated temperatures (ref. 6), and the salt stress corrosion of Ti-8Al-1Mo-1V brake-formed sheet (ref. 7). This paper will review some of the work previously reported in the investigation of Ti-8Al-1Mo-1V alloy sheet as well as current results being obtained from exposures up to 6400 hours at 400° F to 600° F (477° K to 589° K).

The units used for the physical quantities defined in this paper are given both in U.S. Customary Units and in the International System of Units (SI) (ref. 8). Appendix A presents factors relating these two systems of units.

## EXPERIMENTAL PROCEDURES

### Material

In the first phase of the investigation (ref. 4), the Ti-8Al-1Mo-1V alloy sheet (0.040 in. (0.102 cm) thick) was in the single or mill annealed condition; this consisted of an 8-hour anneal at 1450° F (1061° K) with a furnace cool to room temperature. In the latter phases of the investigation (refs. 5 and 7), the Ti-8Al-1Mo-1V alloy sheet (0.050 in. (0.127 cm) thick) was in the duplex anneal condition; this consisted of an 8-hour anneal at 1450° F with a furnace cool to room temperature which was followed by 15 minutes at 1450° F with subsequent air cooling.

### Specimens

The self-stressed specimen and the residual stress specimens are shown in figure 1. Details of the fabrication and cleaning processes are given in references 4 and 7, respectively.

The fabrication process for the self-stressed specimen in the duplex anneal condition differed from that for the mill anneal material, as the former was deburred by a vibratory cleaning process whereas the latter was deburred by hand filing. As the vibratory cleaning treatment was subsequently found to be effective in preventing stress corrosion (see last section of paper), it was necessary to eliminate this effect in order to study the stress corrosion characteristics. Etching 1/2 mil (0.013 mm) from each surface proved sufficient for this purpose.

The construction of the self-stressed specimen (fig. 1(a)) involves machining 1/4- by 4-in. (0.63 by 10.16 cm) strips, bending up the ends of each strip to some predetermined bend angle, and clamping and spotwelding the ends together so as to induce a uniform curvature in the specimen. Bending stresses corresponding to this curvature are determined from geometrical and stress-strain relationships using the measured value of the distance  $d$  between the strips (ref. 9). Stresses are increased by increasing the bend angle of the strip which increases  $d$ . The self-stressed specimens were designed for stresses of 15, 25, 50, and 100 ksi (104, 172, 345, and 690 MN/m<sup>2</sup>, respectively). The nominal thickness  $t$  was either 0.040 or 0.050 in. (0.102 or 0.127 cm).

The residual stress specimens (fig. 1(b)) were brake formed from 0.050-in. (0.127 cm) thick sheet. The top specimen, representative of a typical stiffener section with a right-angle bend (ref. 7), was formed at approximately 200° F (366° K) to a 1/8-in. (0.32 cm) radius. The bottom specimen was brake formed at room temperature over dies with  $\frac{1}{4}$ -,  $\frac{1}{2}$ -, 1-,  $1\frac{1}{2}$ -, and  $2\frac{1}{2}$ -in. radii (0.63, 1.27, 2.54, 3.81, and 6.35 cm, respectively) to an initial bend angle of 90° or the maximum angle (less than 90°) obtainable. The final bend angle varied with the amount of springback which is a function of the radius; the specimen length was kept constant. The residual tensile stress is a maximum on the inside of the bend in the formed specimens. The calculated residual tensile stresses varied from about 70 ksi (483 MN/m<sup>2</sup>) for a 1/8-in. radius to 25 ksi (172 MN/m<sup>2</sup>) for a  $2\frac{1}{2}$ -in. radius.

### Salt Coating and Temperature Exposure

The salts included in the investigation were NaCl, CaCl<sub>2</sub>, MgCl<sub>2</sub>, 7 parts NaCl - 1 part MgCl<sub>2</sub>, and sea water from the mouth of the Chesapeake Bay.

Salt coating techniques were varied during the investigation to include thick as well as thin coatings. The thick coating was applied by dipping the specimen into a boiling, supersaturated salt solution and drying it in an oven at 250° F (394° K). This procedure was repeated until the desired thickness of about 0.005 in. (0.0123 cm) was obtained. To provide thin coatings, specimens were dipped in a 3.4 percent salt solution at ambient temperature and then dried in an oven. In another method, a small drop of the salt solution was placed on the tension surface and allowed to dry.

The salt-coated specimens, as well as uncoated specimens, were exposed in ovens at 400° F to 600° F (477° K to 589° K). With one exception, all ovens were the air-circulating type. Specimens were removed from the ovens after selected exposures for testing at room temperature. One set of residual stress specimens was thermally cycled between room temperature and 550° F (561° K) with cycles from 2 to 16 hours. The cycle time is taken as the elapsed time from specimen insertion to specimen removal from the hot oven.

### Mechanical Tests

The amount of the salt stress corrosion cracking which occurred during the elevated-temperature exposure was determined from compression and bend tests at room temperature of the self-stressed and residual stress specimens, respectively. The behavior of the uncoated specimens during testing is illustrated in figure 2. The uncoated specimens exhibit considerable shortening and bend deflection (figs. 2(a) and 2(b), respectively). Shortening is defined as the decrease in length of the self-stressed specimen and bend deflection as the deflection of the residual stress specimen in the loading direction.

The schematic (fig. 3) illustrates the type of data obtained in the room-temperature compression test of self-stressed specimens and the effect of stress corrosion cracking on the shortening. The uncoated specimen develops the maximum shortening  $\delta_0$  possible with or without fracturing (see dashed line). If stress corrosion cracking occurs in the salt coated specimens (see solid lines), the specimen will fracture and the shortening  $\delta$  is reduced - the longer the exposure and the more the cracking, the less the shortening. The shortening  $\delta_0$  of the uncoated specimen is unaffected by the exposure if the material is stable. If the material is not stable, the shortening of the uncoated specimen may be affected. The ratio  $\delta/\delta_0$  of the shortening of the salt-coated specimen to that for the uncoated or control specimen which has been exposed for the same time is a measure of the stress corrosion damage. In this paper, the effects of the stress corrosion are shown in terms of the relative shortening or relative deflection for the self-stressed and residual stress specimens, respectively.

### Metallurgical Examination

Metallurgical examinations were made to determine the number and characteristics of the stress corrosion cracks. Samples were edge mounted, polished, and etched with a solution of 97 percent  $H_2O$ , 2 percent  $HNO_3$ , and 1 percent  $HF$ , by volume. Crack penetration was measured with a filar micrometer eyepiece in a bench microscope in order to correlate stress corrosion cracking with the mechanical tests.

### Identification of Corrosion Products

An attempt was made to identify the corrosion products by X-ray diffraction. In order to obtain a sufficient amount of corrosion products for this study, a salt-coated coupon of Ti-8Al-1Mo-1V alloy sheet (single anneal) was exposed at  $1000^\circ F$  ( $811^\circ K$ ) for 100 hours. After exposure, the coupon was placed immediately in the X-ray diffractometer and scanned at  $2^\circ$  per minute. Copper  $K_\alpha$  radiation was used (ref. 5).

### ENVIRONMENTAL FACTORS

One phase of the investigation was concerned with the effects of various environmental factors such as the type of salt, coating thickness and type of stress, and oxygen, air pressure, moisture, and surface conditions. Some results of these experiments follow.

#### Effects of Various Chlorides

The relative effect of  $NaCl$ ,  $CaCl_2$ ,  $MgCl_2$ , sea salt, and 7 parts  $NaCl$  - 1 part  $MgCl_2$  (by weight) on the salt stress corrosion of Ti-8Al-1Mo-1V alloy sheet (duplex anneal) at  $600^\circ F$  ( $589^\circ K$ ) is shown in figure 4. Of the

various kinds of salt, NaCl consistently proved more corrosive than 7 parts NaCl - 1 part  $MgCl_2$ , or sea salt. A few tests using  $CaCl_2$  (fig. 4(b)) produced less corrosion than with sea salt, while  $MgCl_2$  caused even less stress corrosion.

In order to determine whether the salt air environment at Langley might be sufficient to cause salt stress corrosion at elevated temperatures, a few self-stressed specimens (100 ksi,  $690\text{ MN/m}^2$ ) in the mill anneal condition, which had been left outdoors for 18,000 hours, were subsequently exposed for 2600 hours at  $550^\circ\text{ F}$  ( $561^\circ\text{ K}$ ). The room-temperature compression tests indicated that an appreciable amount of stress corrosion had occurred in some of the specimens. The amount or type of salt pickup is unknown; as the location was about 2 miles (3.2 km) from the water and there is no industry in the area, the corrosion was probably due to NaCl.

#### Effect of Coating Thickness and Type of Stress

The effect of "thin" and "thick" salt coatings on the stress corrosion of Ti-8Al-1Mo-1V alloy sheet (duplex anneal) at  $550^\circ\text{ F}$  ( $561^\circ\text{ K}$ ) is shown in figure 5 for the self-stressed and residual stress specimens. For a given coating thickness, the results obtained with the residual stress specimen (1/8-in. (0.32 cm) radius) are comparable to those for the self-stressed specimen (50 ksi,  $345\text{ MN/m}^2$ ). Stress corrosion caused by thin coatings occurred much sooner than for thick coatings. After longer times, however, the severity of the stress corrosion was about the same for both types of coatings. Consequently, a thin salt coating can cause severe stress corrosion damage and the manner in which the stress is induced is not a factor. A possible explanation for the greater severity of the stress corrosion with thin coatings may be the greater availability of oxygen.

#### Effect of Oxygen, Air Pressure, Moisture, and Surface Conditions

Various experiments were carried out in an attempt to determine the effect of specific environmental factors on salt stress corrosion of Ti-8Al-1Mo-1V alloy sheet (duplex anneal). The effects of oxygen, air pressure, moisture, and surface conditions were studied (ref. 5) by encapsulating self-stressed specimens in glass tubes containing the desired environment, exposing the tubes to  $550^\circ\text{ F}$  ( $561^\circ\text{ K}$ ) for periods up to 3000 hours, and then testing the specimens at room temperature in the manner previously described.

Oxygen has been recognized as an important factor in the stress corrosion of titanium alloys (for example, refs. 10 and 11) and this is borne out by the results shown in figure 6(a). Tests with nitrogen containing 0.1 percent oxygen show much less corrosion than those with 100 percent oxygen or with air (21 percent oxygen). Some tests were also made to determine the effect of moist chlorine by encapsulating some uncoated specimens in this environment and exposing them at  $550^\circ\text{ F}$  ( $561^\circ\text{ K}$ ). Moist chlorine was found to be very damaging as the specimens failed in the glass tubes within 500 hours, and, consequently, could not be tested in the usual fashion (ref. 5).

The amount of available oxygen decreases at high altitudes because of the reduction in air pressure. Tests at a pressure of  $34$  torr ( $4.0 \text{ kN/m}^2$ ) (equivalent to 70,000-foot (21.3 km) altitude) show much less corrosion than at atmospheric pressure (fig. 6(b)). Tests at  $3 \times 10^{-5}$  torr ( $4.53 \text{ mN/m}^2$ ) (about 380,000-foot (116 km) altitude) show still less corrosion. The decrease in stress corrosion at low pressures is consistent with the reduction in corrosion found for the low oxygen environment. The above experiments under static conditions do not provide any clue as to the possible effect of air flow on stress corrosion. This effect may be important at high altitudes as this could perhaps compensate for the deficiency of oxygen.

Tests with moist and dry air (dewpoints of  $60^\circ \text{ F}$  and  $-40^\circ \text{ F}$  ( $289^\circ \text{ K}$  and  $233^\circ \text{ K}$ )) produced about the same amount of stress corrosion damage (fig. 6(c)). This result may have been due to the fact that the specimens were encapsulated and only a limited amount of moist air was available. Additional tests have subsequently been made by exposing salt-coated specimens in glass tubes through which either dry or moist air was slowly circulated. The tubes extended through a furnace and were heated so as to obtain a specimen temperature of  $600^\circ \text{ F}$  ( $589^\circ \text{ K}$ ). The air flow was maintained at about  $0.3 \text{ cfh}$  ( $0.0085 \text{ m}^3/\text{hr}$ ). Results to date indicate somewhat more corrosion damage in flowing moist air than in flowing dry air. The effect of moisture is still under investigation.

Tests to determine the effect of thin titanium oxide films (fig. 6(d)) employed specimens with polished, as-received, and anodized surfaces having oxide layers from 12 to 16, 100 to 200, and 300 to 600 angstroms thick, respectively. These thin oxide films had little effect on the rate and amount of stress corrosion cracking.

#### Effect of Cyclic Heating and Air Velocity

An attempt was made to determine the effect of cyclic heating (see ref. 7) on the salt stress corrosion of Ti-8Al-1Mo-1V alloy sheet (duplex anneal) from tests of salt-dipped residual stress specimens ( $1/8$ -in. ( $0.32 \text{ cm}$ ) radius) which were thermally cycled between room temperature and  $550^\circ \text{ F}$  ( $561^\circ \text{ K}$ ). The effects of cyclic heating are shown in figure 7 in terms of the accumulated exposure time at  $550^\circ \text{ F}$ . The cycles include 2, 4, 6, 8, and 16 hours at temperature. Examinations of these results reveal that stress corrosion cracking is less severe for short than for long thermal cycles. The results for the 16-hour cycle approach the lower limit of those obtained for constant-temperature conditions. Thus, the stress corrosion obtained from short thermal cycles appears to be considerably less than for constant temperature conditions, and there is some indication that a nucleation period for stress corrosion may be involved.

An attempt was also made to determine the effect of air velocity. In the first experiment, salt-coated self-stressed specimens ( $50 \text{ ksi}$ ,  $345 \text{ MN/m}^2$ ) were exposed in a heater duct at  $650^\circ \text{ F}$  ( $616^\circ \text{ K}$ ) for 48 hours to an air velocity of  $21 \text{ mph}$  ( $9.4 \text{ m/s}$ ). These tests showed no discernible effects of this velocity. In the second experiment, self-stressed specimens with thick and thin salt coatings were tested in the Langley 9- by 6-foot thermal structures tunnel. The specimens were subjected to two 25-second runs at Mach 3 with the specimen

temperature reaching 460° F (511° K). The effect of the two cycles at Mach 3 on the thick salt coatings is illustrated in figure 8. Most of the salt coating was blown away, but enough salt remained to cause stress corrosion cracking after the specimens were exposed for 95 hours in an oven at 600° F (589° K).

#### RELATIVE SUSCEPTIBILITY AND THRESHOLDS

The relative susceptibility of Ti-8Al-1Mo-1V alloy sheet (single anneal) to salt stress corrosion at 550° F (561° K) is shown by comparison with two other titanium alloys in figure 9(a) (from ref. 4). The specimens were of the self-stressed type (100 ksi, 690 MN/m<sup>2</sup>) with heavy salt coatings. Ti-8Al-1Mo-1V undergoes severe stress corrosion cracking within 2000 hours. Ti-6Al-4V is considerably more resistant for the first several thousand hours. Ti-4Al-3Mo-1V was not susceptible after 7000 hours at 550° F but some stress corrosion cracking was obtained after 15,000 hours exposure. As far as salt stress corrosion at elevated temperatures is concerned, Ti-8Al-1Mo-1V is decidedly inferior to Ti-6Al-4V and Ti-4Al-3Mo-1V.

Temperature and stress thresholds for Ti-8Al-1Mo-1V alloy sheet (duplex anneal) in the 400° F to 600° F (477° K to 589° K) range for self-stressed specimens are shown in figure 9(b) for exposures of 3,200 and 5,000 hours, and stresses from 15, 25, and 50 ksi (103, 172, and 345 MN/m<sup>2</sup>). The temperature threshold for salt stress corrosion for stresses of 15 and 25 ksi after 5000 hours exposure appears to be about 500° F (533° K), but for a stress of 50 ksi, the threshold is only slightly above 400° F. Considerably more stress corrosion was obtained after 5000 hours than after 3200 hours exposure at 50 ksi. However, some stress corrosion was also found after 3200 hours at 50 ksi at 450° F (505° K) so that the temperature threshold is also somewhere between 400° F and 450° F for this exposure. The susceptibility of this alloy to salt stress corrosion was found to be about the same for both the single and duplex anneal conditions (refs. 4 and 5).

Temperature and stress thresholds for residual stress specimens (duplex anneal) for exposures up to 6400 hours at 400° F to 600° F (477° K to 589° K) are shown in figure 10(a). The specimens had radii of 1/4, 1/2, and 1 inch (0.63, 1.27, and 2.54 cm), and the corresponding calculated residual tensile stresses were 66, 61, and 56 ksi (456, 422, and 387 MN/m<sup>2</sup>). These preliminary results also point to a temperature threshold slightly above 400° F for exposures up to 6400 hours. No stress corrosion was obtained at 400° F regardless of the magnitude of the exposure or the stress investigated. An exposure of 6400 hours resulted in considerably more corrosion than that for 3200 hours at temperatures above 400° F. These results and those discussed above for self-stressed specimens indicate that salt stress corrosion cracking can be expected for exposures of 3200 hours or more at temperatures slightly above 400° F if the stresses are sufficiently high.

As a tensile stress is required for stress corrosion cracking to take place, the possibility of eliminating stress corrosion in the residually stressed specimens by a conventional stress relief procedure for Ti-8Al-1Mo-1V



alloy sheet (duplex anneal) was investigated. After forming, the 1/4-in. (0.63 cm) radius specimens were stress relieved at 1450° F (1061° K) for 1 hour in either argon or air, and then cooled in argon or air, respectively. The oxide layer for specimens treated in air was removed by blasting with a fine aluminum oxide grit, followed by etching. All specimens were given a salt dip and then exposed up to 2000 hours at 600° F (589° K). The results (fig. 10(b)) indicate that the stress relief treatment in argon was relatively effective in reducing corrosion, but that the treatment in air was unsatisfactory. The fact that the stress relief treatment in argon was effective but that in air was not points to the possibility that contamination due to oxygen or other surface diffusion may be responsible for the increased susceptibility to stress corrosion. Earlier attempts to reduce or eliminate stress corrosion by stress relief treatments in air (ref. 7) were also unsuccessful.

### STRESS CORROSION CRACKS

The stress corrosion cracks observed in both the self-stressed and residual stress specimens of Ti-8Al-1Mo-1V alloy sheet (duplex anneal) exhibited similar characteristics as shown in figure 11. Cracks in both specimens nucleated at the surface and propagated in the direction perpendicular to the tensile stress. In some cases, the cracks were nucleated in the gray areas of general corrosion, while in others the cracks were nucleated in areas which were completely free of corrosion products or pits. Stress corrosion cracks in both types of specimens were intergranular.

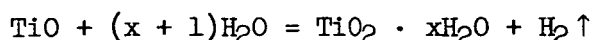
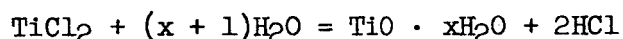
The correlation of the results of the bend tests of the residual stress specimens (1/8-in. (0.31 cm) radius) with the crack penetration measurements is illustrated in figure 12 for exposures up to 500 hours at 550° F (561° K). An increase in the parameter (1-relative deflection) corresponds directly to the increase in the maximum crack depth. A correlation between relative shortening in the compression test of self-stressed specimens with the crack penetration had been noted previously (ref. 4). Consequently, the shortening and deflection measurements in the tests of the self-stressed and residual stress specimens provide quantitative information on the amount of the stress corrosion cracking.

### CORROSION PRODUCTS AND MECHANISM

#### Identification of Corrosion Products

The results of the X-ray diffraction analysis of corrosion products formed at 1000° F (811° K) on Ti-8Al-1Mo-1V alloy sheet (single anneal) are shown in table I. The experimentally determined d-spacings along with their estimated relative intensities are compared with the d-spacings listed in the ASTM index for TiO<sub>2</sub>, TiO, and TiCl<sub>2</sub>. The d-spacings for Ti and NaCl were omitted from the table. The X-ray diffraction data provide strong evidence toward TiO<sub>2</sub> (rutile) as a corrosion product, while the identification of TiO<sub>2</sub> (anatase),

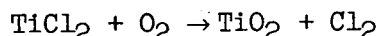
•  $\text{TiO}$ , and  $\text{TiCl}_2$  may be questioned. However, two further observations indicate that  $\text{TiCl}_2$  was actually present in the corrosion products. First, when the corrosion products were dissolved in water, a gas bubbled off and the solution became slightly acidic. Kochka and Peterson (ref. 11) observed a similar reaction and believed it to be caused by the combination of  $\text{TiCl}_2$  and water, as follows:



Second, the peaks identified as  $\text{TiCl}_2$  decreased with time at room temperature and disappeared after the sample was lightly washed in distilled water. This would be expected since  $\text{TiCl}_2$  is hygroscopic and decomposes in water. Therefore,  $\text{TiCl}_2$  appears to be one of the principal corrosion products at  $1000^\circ \text{F}$  and may be the key product at temperatures below about  $600^\circ \text{F}$  ( $589^\circ \text{K}$ ).

#### Mechanism

Many investigators have proposed theories for the stress corrosion cracking in titanium alloys where  $\text{TiCl}_2$  is a key corrosion product. One of the difficulties in verifying these mechanisms has been the problem of positively identifying  $\text{TiCl}_2$  in the corrosion products. This might be expected since  $\text{TiCl}_2$  would be oxidized to form  $\text{Cl}_2$  as follows:



The chlorine would then react with the titanium, initiating stress corrosion cracks and forming more  $\text{TiCl}_2$ . If this type of cyclic reaction occurs at  $500^\circ \text{F}$  to  $600^\circ \text{F}$  ( $533^\circ \text{K}$  to  $589^\circ \text{K}$ ), the amounts of  $\text{TiCl}_2$  actually present at any instant would be too small to pick up with normal X-ray diffraction techniques. It is believed that the reaction at  $1000^\circ \text{F}$  ( $811^\circ \text{K}$ ) produced enough  $\text{TiCl}_2$  to enable the identification.

Before accurate chemical equations describing the mechanism can be formulated, the remaining corrosion product or products must be identified. Table I shows 12 unidentified diffraction peaks. These peaks may be diffracted from some complex sodium-titanium compound which has not yet been cataloged in the ASTM index.

#### PROTECTIVE TREATMENTS

Various protective treatments such as shot peening, nickel plating, vibratory cleaning, and polyimide coatings were investigated in order to determine

their relative effectiveness in reducing salt stress corrosion at 600° F (589° K). These preliminary experiments (see fig. 13) were carried out with a small number of residual stress specimens with a 1/4-in. (0.63 cm) radius which were given a thin salt coating after the surface treatment. Considerable work still needs to be done to determine the durability and practicability of the various treatments.

Shot peening was accomplished after brake forming with glass particles (SiO<sub>2</sub>) 44 to 70 microns in diameter. The peening consisted of a 10-second treatment with a pressure of 60 psi (414 kN/m<sup>2</sup>). The amount of the residual surface stresses introduced by this process is unknown. Shot peening was effective in preventing salt stress corrosion cracking for exposures up to about 1000 hours at 600° F (589° K). Shot peening with larger particles (250 to 500 microns diameter) was slightly less effective. The effectiveness of the treatment apparently drops off at the longer exposures (see fig. 13).

Some of the specimens were electroplated with a nickel coating about 1/3 to 1/2 mil (0.008 to 0.013 mm) thick after forming. The nickel plating proved effective in preventing stress corrosion up to 2000 hours at 600° F (589° K). The adherence of the nickel plating under cyclic loading conditions, however, requires investigation.

The vibratory cleaning treatment is a commercial cleaning and deburring process in which the specimen is vibrated in a bath containing aluminum oxide triangles (3/8 in. (0.95 cm) sides, 1/4 in. (0.63 cm) thick) and a cleaning solution. The bath was vibrated at 1450 cmp (24 Hz) for 8 hours. This treatment also proved effective in preventing stress corrosion (fig. 13). As previously mentioned, the efficacy of this method was inadvertently discovered when the process was used for deburring the strips used in the fabrication of the self-stressed specimens. These self-stressed specimens (50 ksi, 345 MN/m<sup>2</sup>) showed no evidence of salt stress corrosion after 6400 hours exposure at 600° F (589° K).

Other specimens were coated with an organic polymer - a commercial aromatic polyimide. The coatings were 1/2 to 1 mil (0.013 to 0.025 mm) thick. The results of the exposure (fig. 13) indicate that such coatings can be effective up to about 1000 hours at 600° F (589° K). The coatings tended to degrade and peel off after this exposure, however, and consequently, would not provide any subsequent protection against salt stress corrosion.

#### CONCLUDING REMARKS

Considerable data have been obtained on the salt stress corrosion of Ti-8Al-1Mo-1V alloy sheet in the 400° F to 600° F (477° K to 589° K) range from tests of many small self-stressed and residual stress specimens. Under constant temperature conditions, it is now evident that the temperature threshold for this susceptible alloy is only slightly above 400° F for a stress of 50 ksi (345 MN/m<sup>2</sup>) and an exposure of 5000 hours. Under cyclic heating conditions for 2- or 4-hour cycles, stress corrosion may be less severe. Of the various salts

investigated, NaCl appears to be the severest corrodent.  $\text{TiCl}_2$  has been identified as a corrosion product at  $1000^\circ \text{F}$  ( $811^\circ \text{K}$ ), but the stress corrosion mechanism is still unknown. The effects of several environmental factors such as oxygen and air pressure seem to be relatively well defined, but the role of moisture and air velocity still requires investigation. Various protective surface treatments such as shot peening, nickel plating, vibratory cleaning, and polyimide coatings were found effective in preventing salt stress corrosion for exposures up to 2000 hours at  $600^\circ \text{F}$  ( $589^\circ \text{K}$ ).

## APPENDIX A

### CONVERSION OF U.S. CUSTOMARY UNITS TO SI UNITS

The International System of Units (SI) was adopted by the Eleventh General Conference on Weights and Measures, Paris, October 1960, in Resolution No. 12 (ref. 8). Conversion factors for the units used herein are given in the following table:

Physical quantity	U.S. Customary Unit	Conversion factor (*)	SI Unit
Length	mile	1.609	kilometer (km)
	foot	0.3048	meter (m)
	inch	2.54	centimeter (cm)
Stress	ksi	6.89	meganewtons per sq meter (MN/m <sup>2</sup> )
Pressure	torr	133.322	newtons per sq meter (N/m <sup>2</sup> )
	psi	6.894	kilonewtons per sq meter (kN/m <sup>2</sup> )
Temperature	(°F + 459.67)	5/9	degrees Kelvin (°K)
Velocity	mph	0.44704	meters per sec (m/s)
Frequency	cpm	0.01667	cycles per sec, hertz (Hz)
Flow rate	cfh	0.0283	cubic meters per hour (m <sup>3</sup> /hr)

\*Multiply value given in U.S. Customary Unit by conversion factor to obtain equivalent value in SI Unit.

Prefixes to indicate multiple of units are as follows:

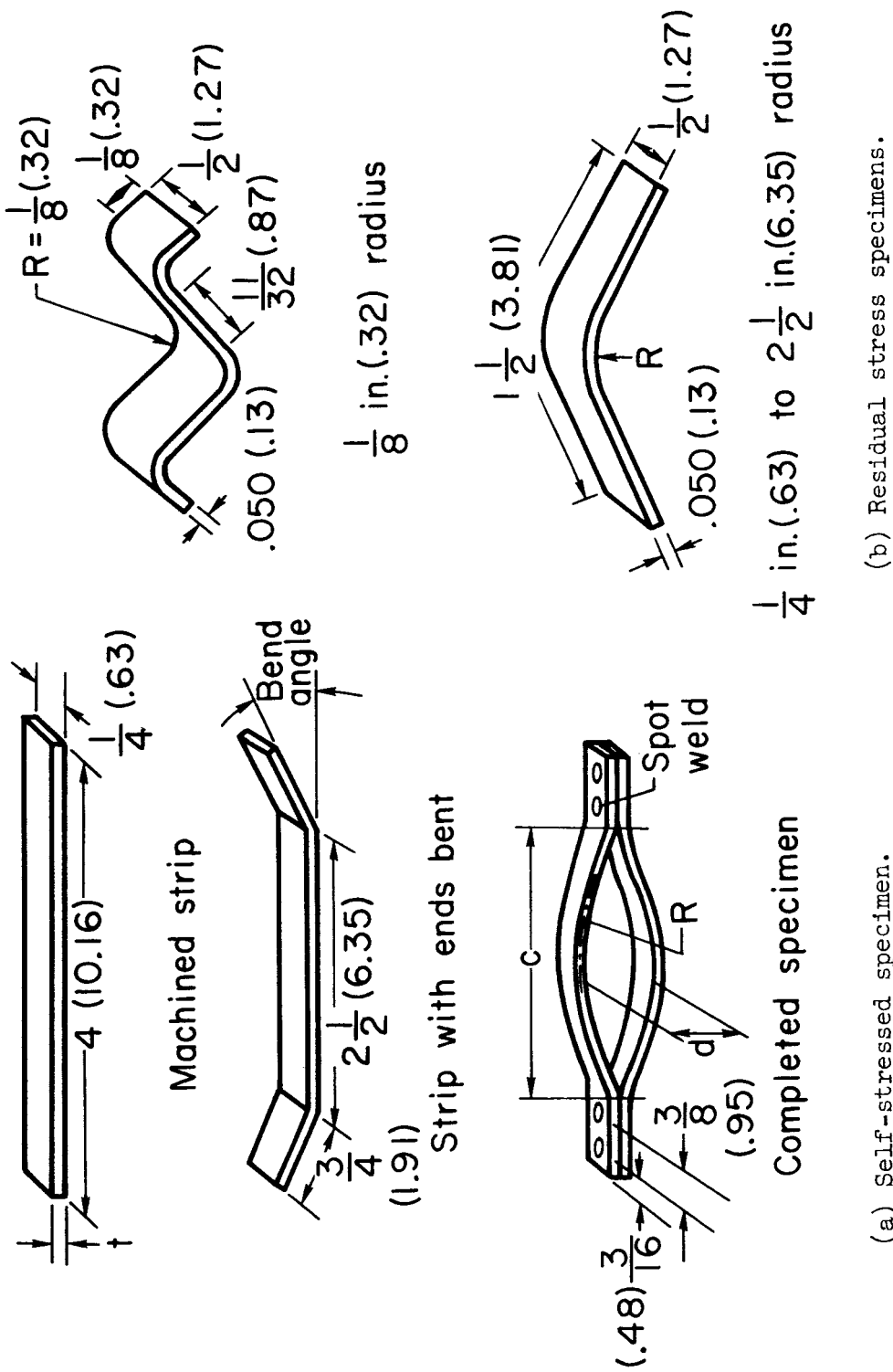
Prefix	Multiple
milli (m)	10 <sup>-3</sup>
centi (c)	10 <sup>-2</sup>
kilo (k)	10 <sup>3</sup>
mega (M)	10 <sup>6</sup>

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TABLE I.- X-RAY DIFFRACTION ANALYSIS OF CORROSION PRODUCTS

Experimental		ASTM index								Unidentified diffraction peaks
		TiO <sub>2</sub> (Rutile)		TiO <sub>2</sub> (Anatase)		TiO		TiCl <sub>2</sub>		
		d-spacing, Å	Relative intensity	d-spacing, Å	Relative intensity	d-spacing, Å	Relative intensity	d-spacing, Å	Relative intensity	
d-spacing, Å	Relative intensity									
4.1639	50									X
3.2663	100	3.245	100					2.68	100	
2.6758	60							2.51	20	X
2.6315	50									
2.5060	15									
2.4953	55	2.489	41							
2.3926	30			2.379	22			2.390	80	
2.3852	10									
2.3075	15	2.297	7							
2.2607	10									
2.1912	15	2.188	22					2.27	20	
2.0562	5	2.054	9							
2.0508	5									
1.9087	5									X
1.8908	5			1.891	33					X
1.8717	30									
1.7807	10							1.86	40	
1.7659	5									
1.7434	10									
1.6885	40									X
1.5690	5	1.687	50	1.699	21			1.68	40	X
1.5363	10									
1.5276	15									
1.4784	10									
1.3810	5	1.480	8	1.480	13			1.53	40	
1.3616	10	1.360	16	1.367	5					X
1.3530	10									
1.3483	10	1.347	7							X
1.3060	10	1.305	1							
1.2226	5									X
1.2112	5									X
1.1493	5	1.1485	4							
1.1128	5									
1.0931	5	1.0933	4					1.14	40	
1.0846	5	1.0827	4	1.0869	3			1.11	40	

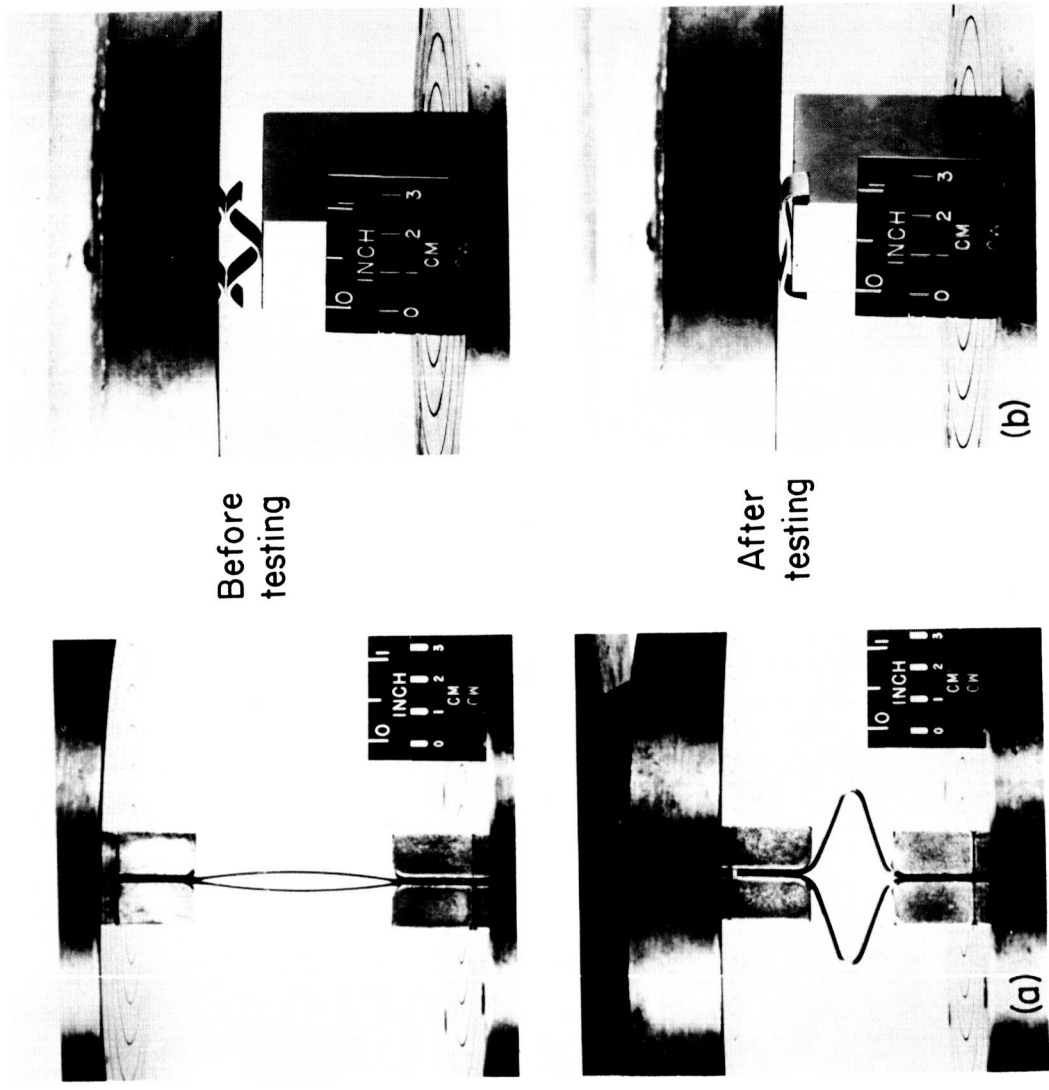


(a) Self-stressed specimen.

(b) Residual stress specimens.

Figure 1.- Construction of the self-stressed specimen and the two types of residual stress specimens. Dimensions in inches with cm in parenthesis.





(a) Self-stressed specimen.

(b) Residual stress specimen.

Figure 2.- Compression test of self-stressed specimen and reverse-bend test of residual stress specimen.

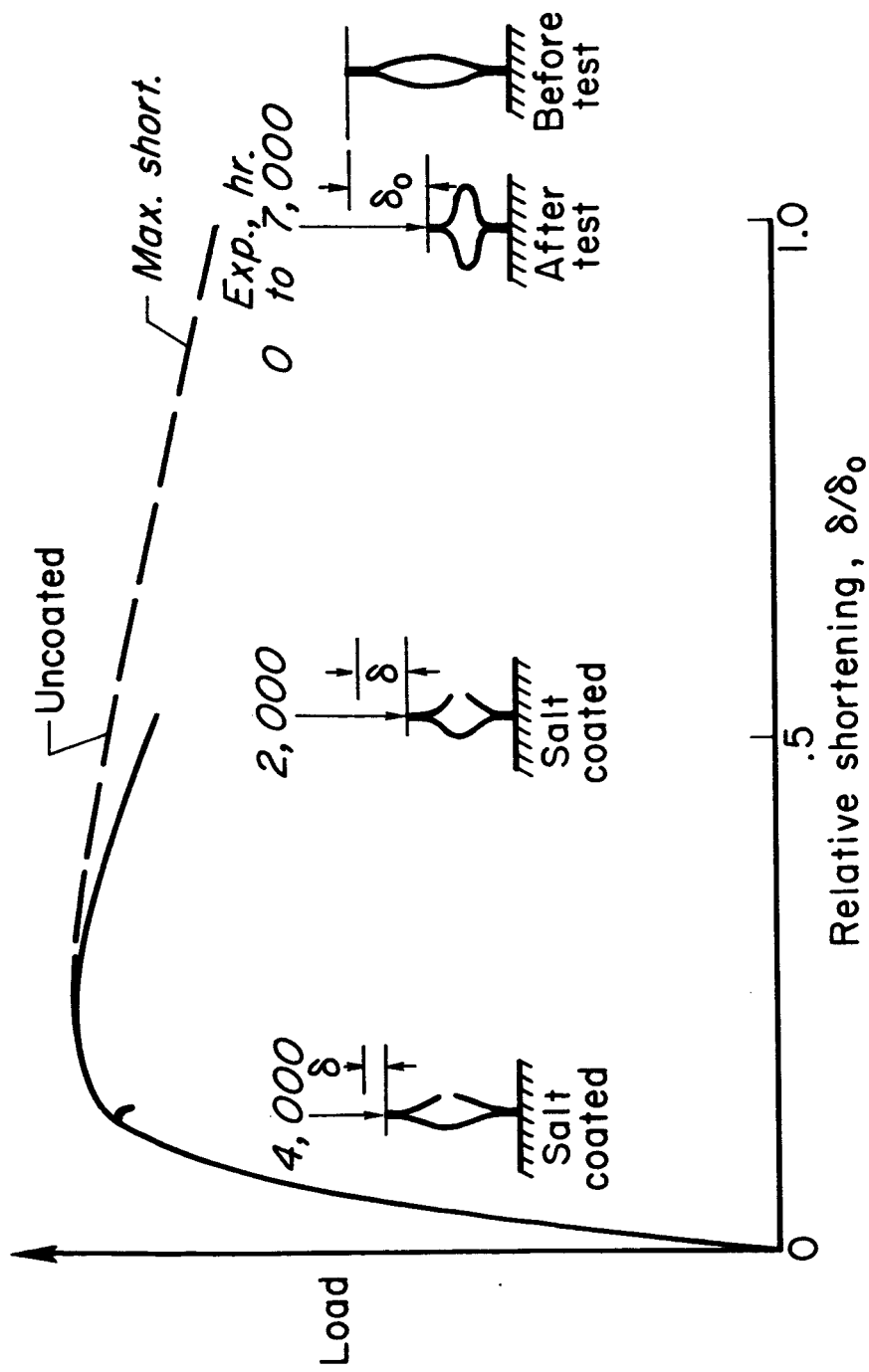
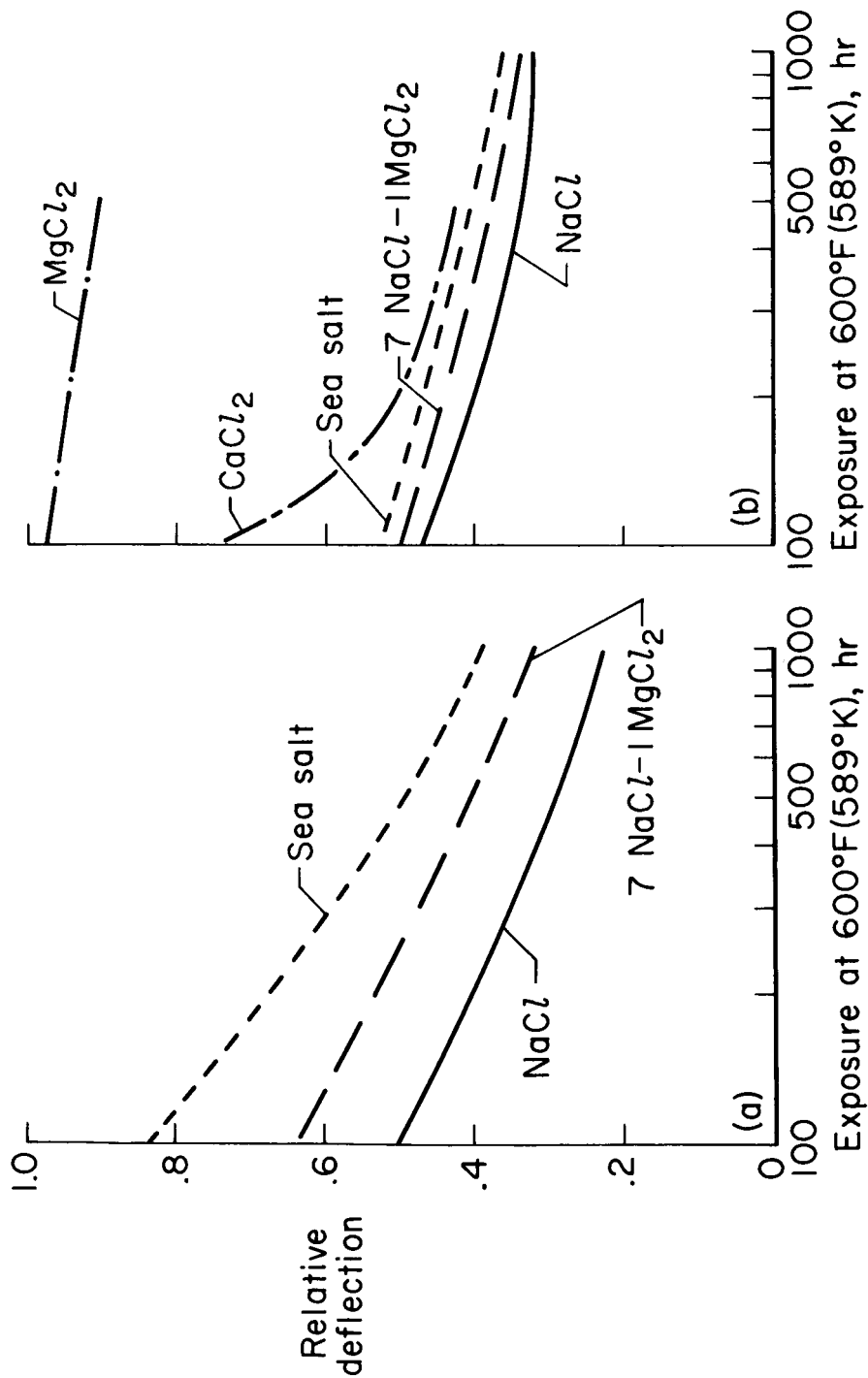


Figure 3.- Schematic of compression test of self-stressed specimen and shortening measurements.



(a) Self-stressed specimen.  
50 ksi (345 MN/m<sup>2</sup>).

(b) Residual stress specimen.  
1/4-in. (0.63-cm) radius.

Figure 4.- Effect of type of salt on stress corrosion of Ti-8Al-1Mo-1V alloy sheet at 600° F (589° K).

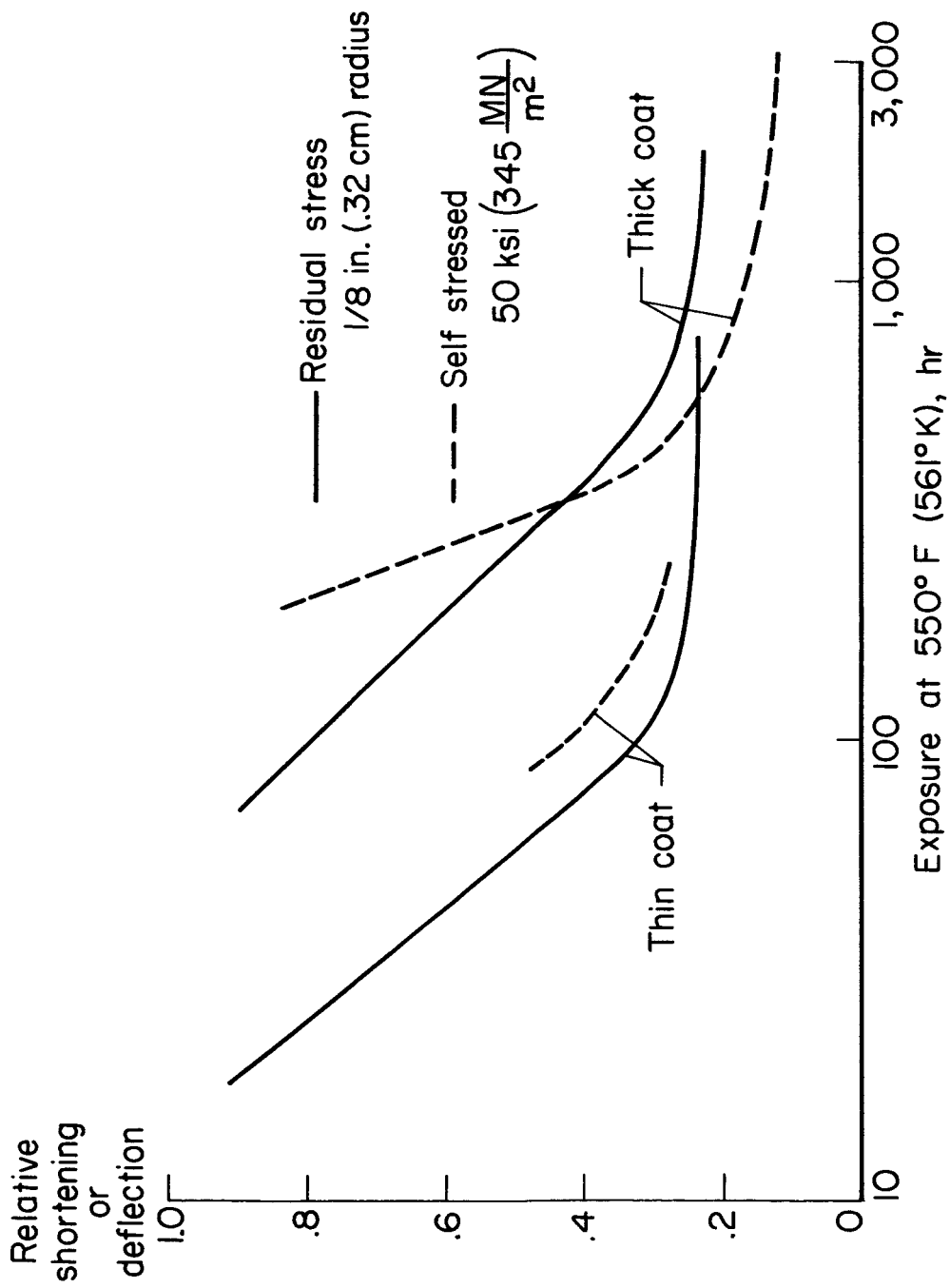


Figure 5.- Effect of salt coating thickness and type of stress on stress corrosion of Ti-8Al-1Mo-1V alloy sheet at 550° F (561° K).

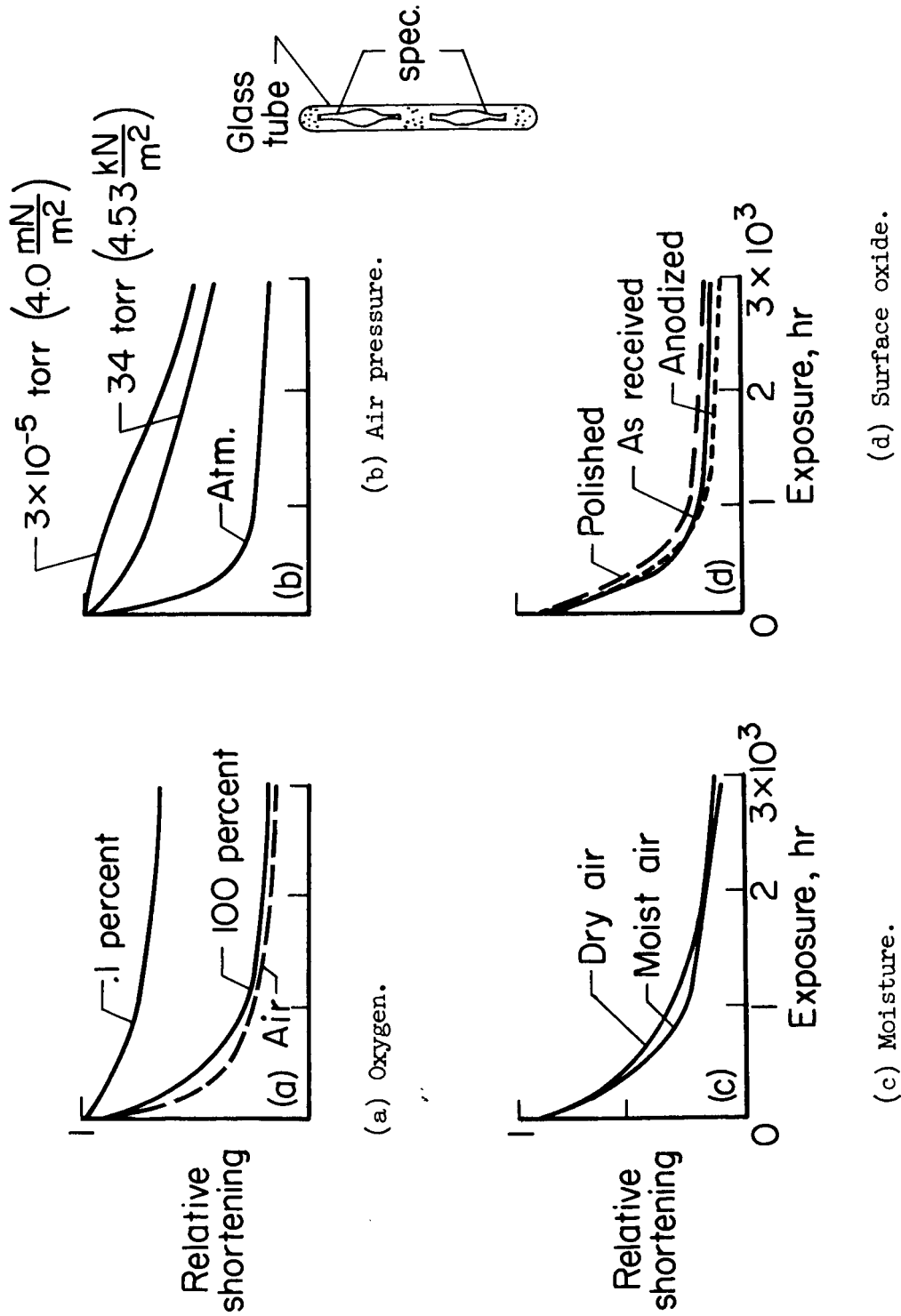


Figure 6.- Effect of oxygen, air pressure, moisture, and surface oxide on salt stress corrosion of Ti-8Al-1Mo-IV alloy sheet at 550° F (561° K).

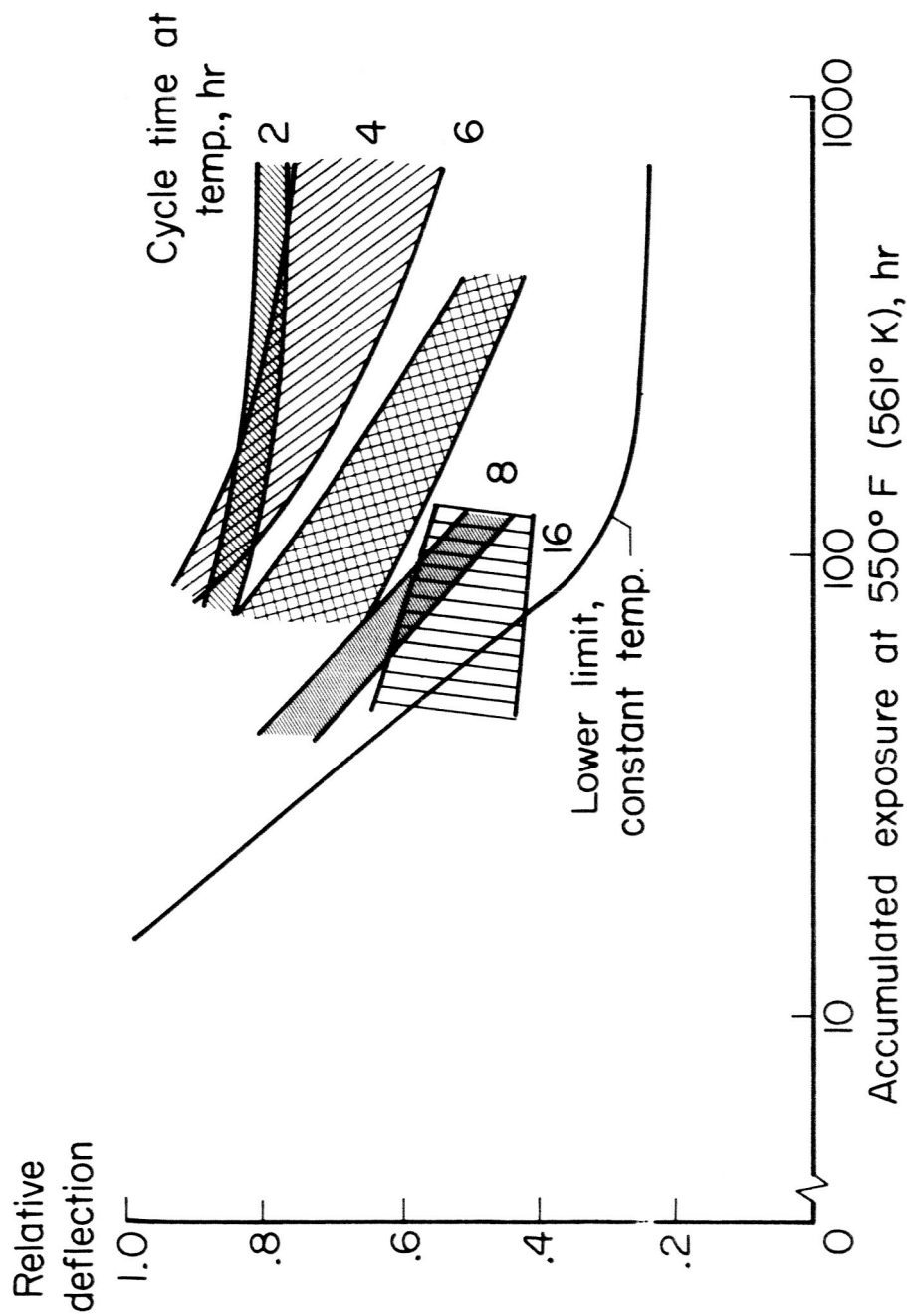
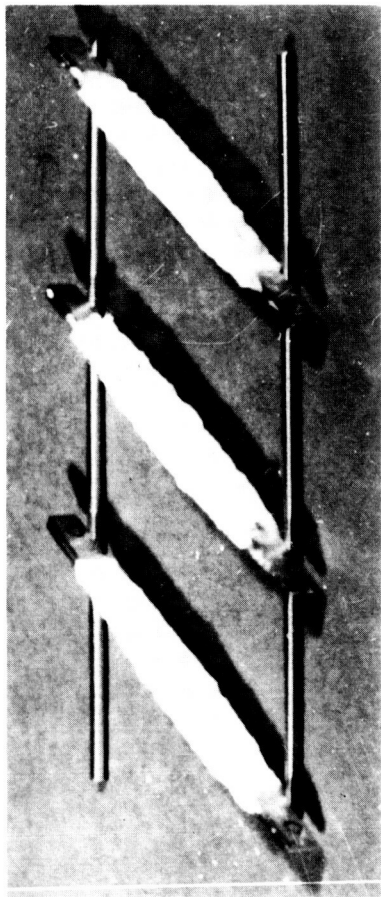
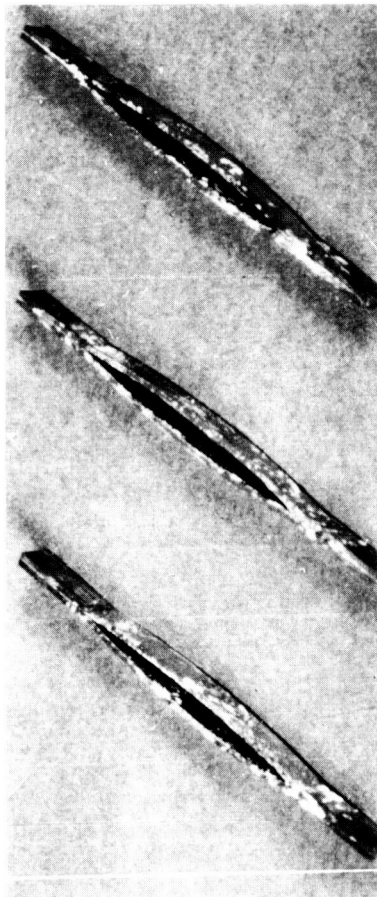


Figure 7.- Effect of 2-, 4-, 6-, 8-, and 10-hour thermal cycles on salt stress corrosion of Ti-8Al-1Mo-1V alloy sheet at 550° F (561° K). (Tests with residual stress specimens with 1/8-in. (0.32-cm) radius.)

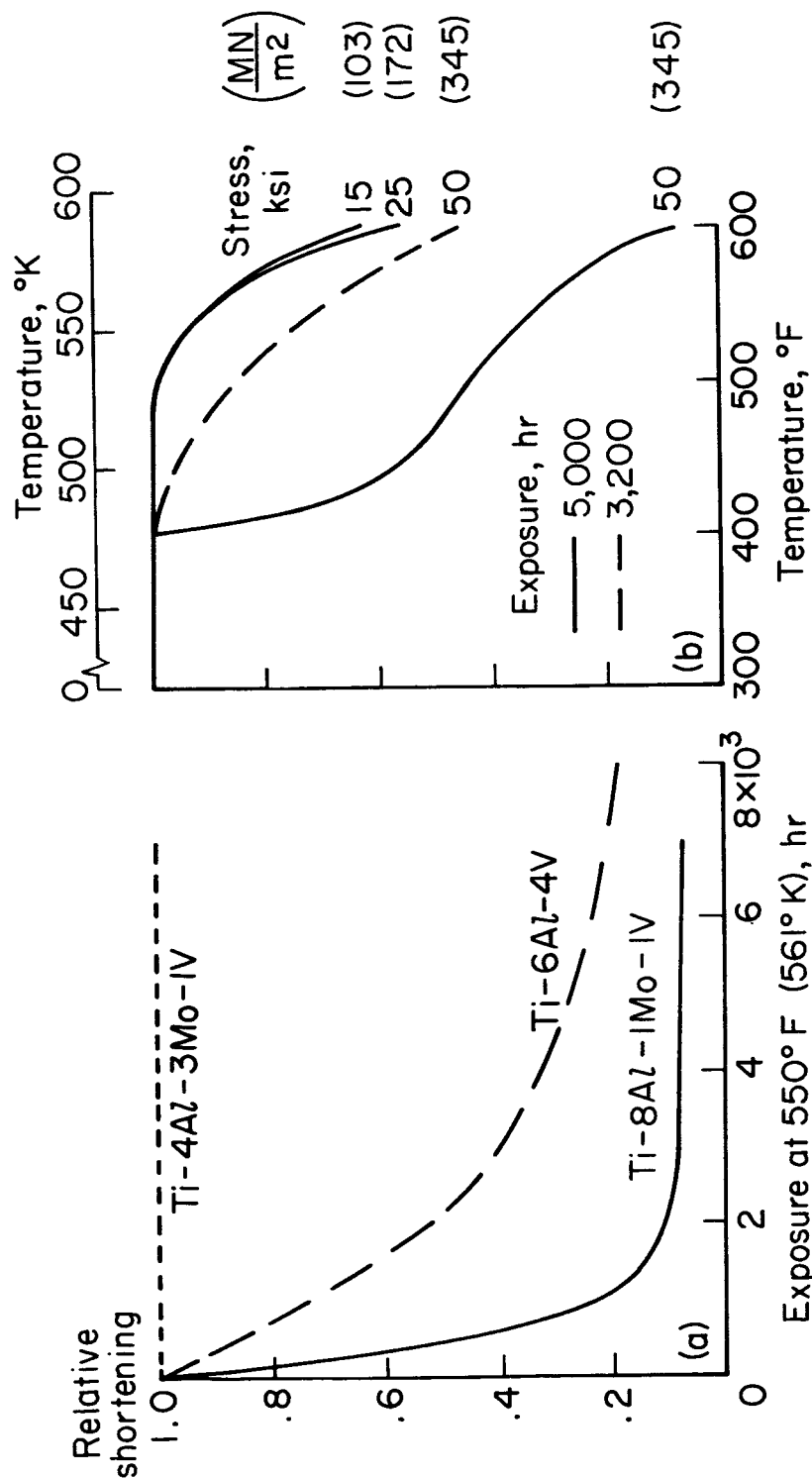


Original coating



Coating after 2 cycles

Figure 8.- Effect of two 25-second cycles at Mach 3 on heavy salt coatings.

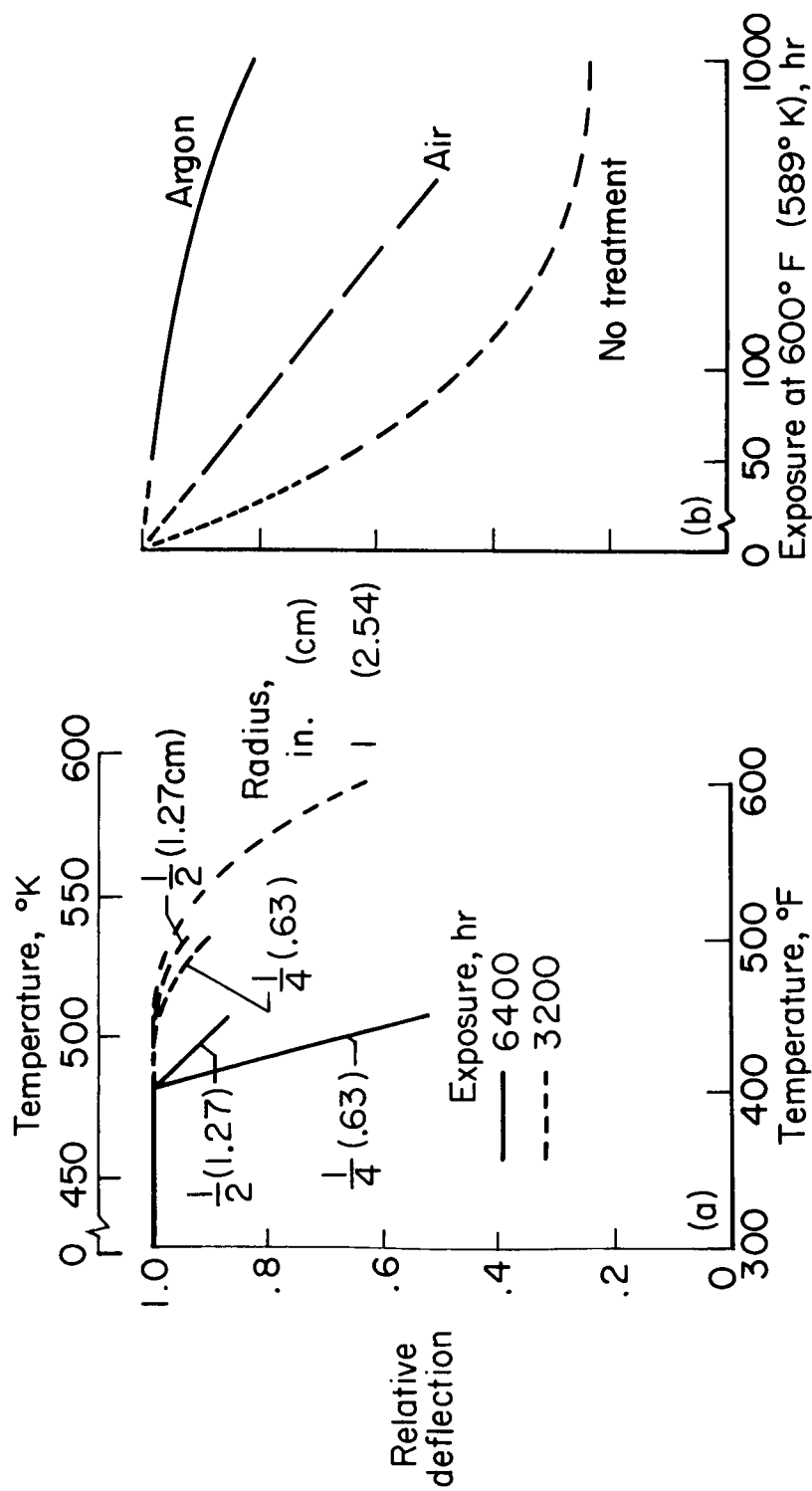


(a) Relative susceptibility.  
100 ksi (690 MN/m<sup>2</sup>.)

(b) Stress-temperature thresholds.

Figure 9.- Relative susceptibility of three titanium alloys to salt stress corrosion at 550° F (561° K), and stress-temperature thresholds for self-stressed specimens of Ti-8Al-1Mo-1V alloy sheet for exposures up to 5000 hours at 400° to 600° F (477° to 589° K).

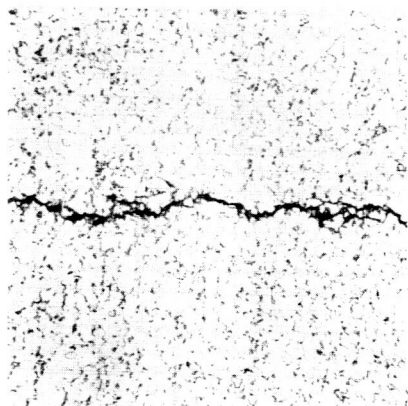




(a) Stress-temperature thresholds.

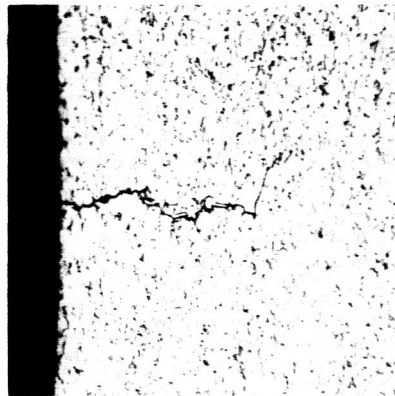
(b) Stress relief treatment.  
1/4-in. (0.63-cm) radius.

Figure 10.- Stress-temperature thresholds for residual stress specimens for exposures up to 6400 hours at 400° to 600° F (477° to 589° K), and the effectiveness of a stress relief treatment consisting of 1 hour at 1450° F (1061° K).

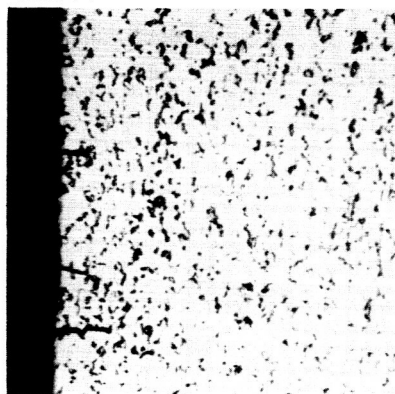


Surface  
view

.002 in.  
└─────────┘  
.0508 mm



Edge  
view



(a) Self-stressed specimen.  
5000 hr at 600° F (589° K).

(b) Residual stress specimen.  
Top: 500 hr at 550° F (561° K)  
Bottom: 100 hr at 550° F (561° K)

Figure 11.- Typical stress corrosion crack in self-stressed and residual stress specimens of Ti-8Al-1Mo-1V alloy sheet.

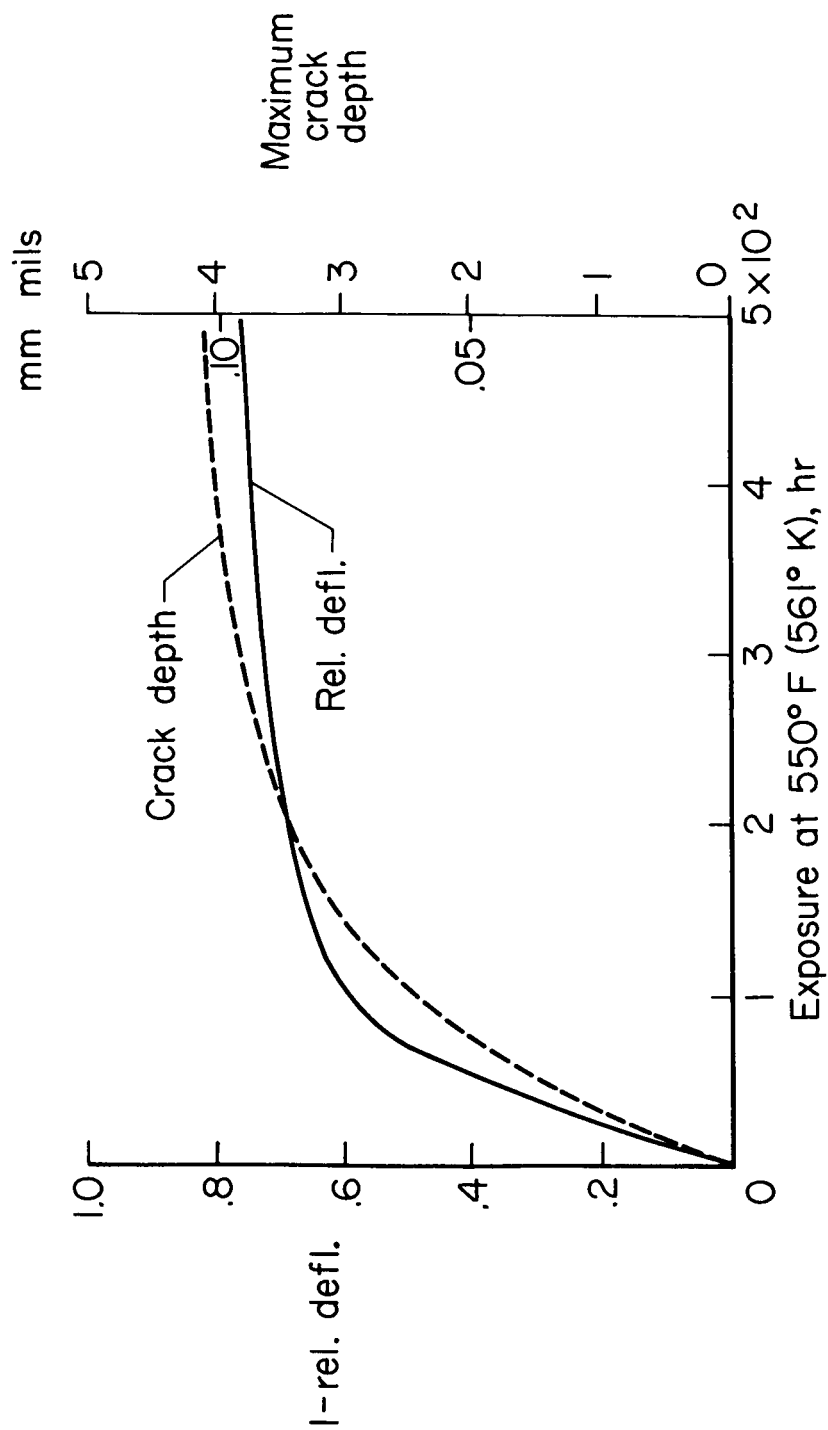


Figure 12.- Correlation of lower limit of relative deflection in reverse-bend tests with maximum crack penetration in Ti-8Al-1Mo-1V alloy sheet for exposures up to 500 hours at 550° F (561° K).

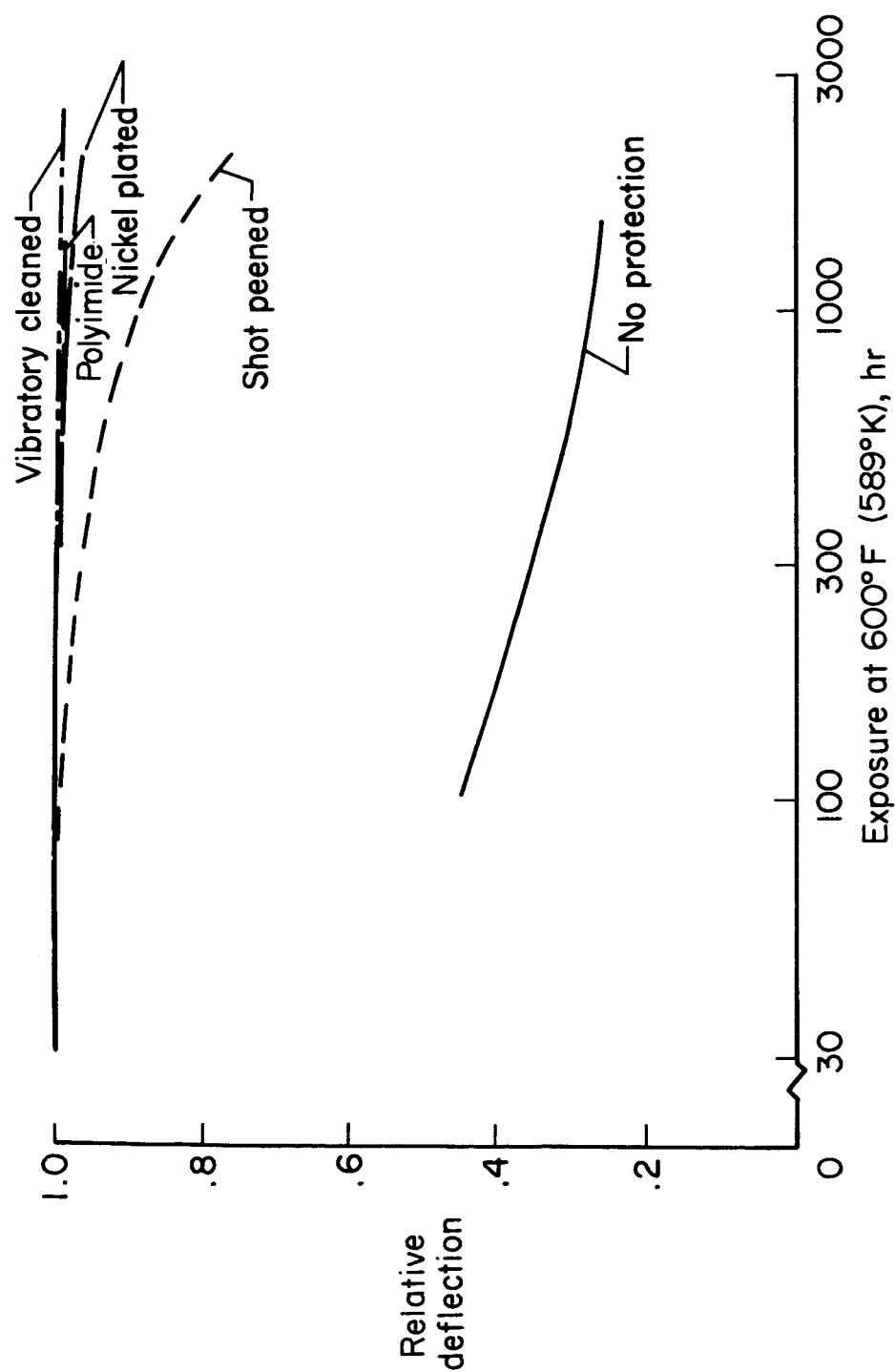


Figure 13.- Effect of various protective treatments on salt stress corrosion of Ti-8Al-1Mo-1V alloy sheet at 600° F (589° K). Tests with 1/4-in. (0.63-cm) residual stress specimens.